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FAB 26

NITROSAMINES

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H. BROOKES
ASSISTANT EDITOR

1

In vivo formation of nitroso compounds. VI. In vitro and in vivo formation of dimethylnitrosamine by bacteria isolated from human saliva.
Ishiwata, H.; Tanimura, A.; Ishidate, M.
Journal of the Food Hygienic Society of Japan [Shokuhin Eiseigaku Zasshi] **17** (1) 59-65 (1976) [22 ref. En] [Nat. Inst. of Hygienic Sci., Kamiyoga 1-chome, Setagaya-ku, Tokyo, Japan]
See FSTA (1976) **8** 7C326 for part V.

2

[Thermoionic detector for chromatographic determination of nitrosamines.]
Wozniak, J.; Tokarz, A.; Miskiewicz, W.
Roczniki Panstwowego Zakladu Higieny **27** (3) 261-268 (1976) [11 ref. Pl, ru, en] [Inst. Biofarmacji Akad. Medycznej, Warsaw, Poland]

3

Determination of a non-volatile nitrosamine by using denitrosation and a chemiluminescence analyser.
Downes, M. J.; Edwards, M. W.; Elsey, T. S.; Walters, C. L.
Analyst **101** (1206) 742-748 (1976) [11 ref. En] [British Food Manufacturing Ind. Res. Ass., Randalls Road, Leatherhead, Surrey KT22 7RY, UK]
A method has been devised for detn. of N-nitrososarcosine, representative of the group of non-volatile nitrosamines of physiological origin, whereby the nitrosamine is denitrosated with hydrogen bromide to form the volatile nitrosyl bromide. After dehalogenation in a heated converter, the nitrosamine can be determined as nitric oxide by using a chemiluminescence analyser, the reponse being linear up to at least 200 µg. The limit of detection is below 6 ng, i.e. much lower than that for detn. of N-nitrososarcosine as inorganic nitrite in solution after denitrosation. A response is also obtained from inorganic nitrite but this can be differentiated from that of N-nitrososarcosine by use of acetic acid alone, prior to denitrosation with hydrogen bromide in the same solvent. AS

4

Effect of storage on the concentration of proline and other free amino acids in pork bellies.
Lakritz, L.; Spinelli, A. M.; Wasserman, A. E.
Journal of Food Science **41** (4) 879-881 (1976) [13 ref. En] [USDA, E. Regional Res. Cent., Philadelphia, Pennsylvania 19118, USA]
Proline is a potential precursor in the formation of N-nitrosopyrrolidine, a carcinogen that has been detected in fried bacon. The concn. of free proline and 11 additional amino acids were measured on the first and eighth day after slaughter and storage at 2°C. The free amino acids (proline, alanine, glycine, isoleucine, leucine, methionine, phenylalanine, tyrosine, valine, glutamic acid, cystine, aspartic acid) were determined in separated lean tissue, in the adipose tissue, and in samples of intact green pork bellies. The concn. of most of the amino acids increased with storage. Proline in the intact pork bellies increased approx. 52% after 1 wk of storage. Over the same period, free proline in the lean and in the adipose tissues increased 50 and 90% respectively. Effects of extended storage (28 days) on the concn. of free proline in lean tissue was also determined. IFT

5

Pathways of formation of N-nitrosopyrrolidine in fried bacon.
Nakamura, M.; Baba, N.; Nakaoka, T.; Wada, Y.; Ishibashi, T.; Kawabata, T.
Journal of Food Science **41** (4) 874-878 (1976) [26 ref. En] [Kanagawa Prefectural Public Health Lab., 52-2, Nakao-cho, Asahi-ku, Yokohama, Kanagawa-ken 241, Japan]
The study was designed: (i) to conduct a commercial bacon survey, with special reference to concn. of residual nitrite (NO_2^-), proline (Pro), pyrrolidine (Pyr), nitrosoproline (NO-Pro) and nitrosopyrrolidine (NO-Pyr) in various brands of bacon collected in areas of Tokyo and Kanagawa Prefecture, with analyses of bacon done before and after frying; (ii) to conduct a quantitative evaluation of the possible pathways of the formation of NO-Pyr from NO-Pro, Pro or Pyr occurring in the actual ground bacon samples at elevated temp.; and (iii) to determine the stability of NO-Pyr in bacon against high temp. heating. In the range 100-150°C with a heating time of 10 min, amounts of NO-Pyr formed from free Pro via Pyr were almost the same as those formed via NO-Pro. On the contrary, at 175°C or above, the yield of NO-Pyr via Pyr was greater than that formed via NO-Pro. In bacon, 80-90% of NO-Pyr disappeared when it was brought to 200°C within 10 min, while NO-Pyr in vitro was fairly stable to heating, with only a 2-5% loss under the same heating conditions. IFT

6

[Nitrosamines in meat products: tests on firm dry sausages made with various additives.] Zum Vorkommen von Nitrosaminen in Fleischerzeugnissen und Untersuchungen an schnittfesten Rohwürsten bei unterschiedlichen Zusätzen.
Kotter, L.; Fischer, A.; Schmidt, H.; Walters, C. L.; Hauser, E.; Heiz, H. J.
Fleischwirtschaft **56** (7) 997-1000, 1003-1004, 1007 (1976) [78 ref. De, en, fr] [Inst. für Hygiene & Tech. der Lebensmittel tierischen Ursprungs der Univ., Veterinärstrasse 13, 8000 Munich 22, Federal Republic of Germany]
The formation of nitrosamines in factory-produced (quickly ripened) dry sausages was investigated. Sausages were made with 30 g nitrite curing salt (NaCl plus 0.5-0.6% $NaNO_2$)/kg and various additives: ascorbic acid or sodium ascorbate, glucono-δ-lactone (GdL), dextrose, or

starter cultures. 10 ppm diethylamine (DEA) was added to parallel batches. Sausages were examined by MS-GLC (after maturing for 12-29 days) for dimethyl nitrosamine (DMNA), diethyl nitrosamine (DENA), nitrosopiperidine (NPi), nitrosopyrrolidine (NPy), and some samples also for nitrosmorpholine (NMo) and methyl ethyl nitrosamine (MENA), pH, NO_2^-, NO_3^- and red colour formation. Results are shown in a table. Given a detection limit of 1-2 parts/billion no DMNA, DENA, NPi or NPy was detected when no DEA was added, and 0.4-3.5 parts/billion DENA was detected in isolated cases when DEA was added. DENA formation was not related to any additive. No other nitrosamine was detected. 3 methods are recommended for controlling nitrosamine formation in foods or in the human stomach: ensuring that the diet is low in nitrosating substances, that foods are low in amines and amine-containing drugs are controlled, and excluding processing methods that favour reactions between nitrosating and nitrosatable substances in foods. Both ascorbic acid and GdL inhibit nitrosamine formation. RM

7

[Relationship between nitrate, nitrite and nitrosamines in plants and soil.] Über Zusammenhänge von Nitrat-Nitrit-Nitrosaminen in pflanzlichem Material und im Boden.
Dressel, J.
Qualitas Plantarum - Plant Foods for Human Nutrition **25** (3/4) 381-390 (1976) [33 ref. De, en] [Landwirtschaftliche Versuchssta. der BASF, 6703 Limburgerhof, Postfach 220, Ludwigshafen/Rhein, Federal Republic of Germany]

An attempt was made to establish whether intensive N dressings lead to the formation of nitrosamines (dimethyl- and diethyl-nitrosamine) in plants. The findings were negative both in cereals (wheat and barley) and in vegetables. In model experiments, both nitrosamines were broken down when added to soil. Degradation appears to be of a microbial nature and to proceed by way of evapotranspiration. Nitrosamines absorbed by plants were detectable for only a very short period. AS

8

Carcinogenic N-nitroso compounds.
Archer, M. C.
Abstracts of Papers, American Chemical Society **172**, AGFD 18 (1976) [En] [Dep. of Nutr. & Food Sci., Massachusetts Inst. of Tech., Cambridge, Massachusetts 02139, USA]

N-nitroso compounds form a group of versatile and potent carcinogens. They may be formed by interaction of nitrous acid with secondary or tertiary amines, secondary amides, ureas, carbamates and guanidines. Since these precursors can occur in foods, N-nitroso compounds may be relevant to the causation of human cancer. Nitrosamines are the most extensively studied N-nitroso compounds. Several factors affect the rate of formation of nitrosamines. These include pH, temp., anions, carbonyl compounds and surfactants. Nitrosamine formation can be inhibited by reducing agents such as ascorbic acid or by other compounds such as phenols which react rapidly with nitrous acid. Nitrosamines can be formed by reaction of nitrite and secondary amines in gastric juice. In contrast to other N-nitroso compounds, nitrosamines exert their carcinogenic effect only after metabolic oxidation. The ultimate carcinogen in probably an electrophilic species which initiates the carcinogenic process by reaction with critical cellular nucleophiles. AS

9

Hydroxyalkyl N-nitrosamines from the nitrosation of spermidine.
Hotchkiss, J. H.; Scanlan, R. A.; Libbey, L. M.
Abstracts of Papers, American Chemical Society **172**, AGFD 63 (1976) [En] [Dep. of Food Sci. & Tech., Oregon State Univ., Corvallis, Oregon 97331, USA]

The naturally occurring polyamine spermidine was reacted with sodium nitrite in an acidic, aqueous medium. After-extraction and solvent removal the products were derivatized to trifluoroacetate (TFA) derivatives and separated by GLC. Low resolution mass spectral data were collected on the GLC effluent. Those peaks which showed characteristic alkyl N-nitrosamine spectra were assigned a structure based upon their spectrum. 2 dihydroxylated alkyl N-nitrosamines were synthesized by an alternate route and retention times and mass spectra of their TFA derivatives compared with compounds derived from nitrosated spermidine. The N-nitrosamines 3-hydroxypropyl(4-hydroxybutyl)N-nitrosamine and 2-hydroxypropyl(3-hydroxybutyl)N-nitrosamine were shown to be present in the nitrosation products of spermidine. NMR and IR data were collected on the 2 nitrosamines. AS

10

Effects of dietary modification on carcinogen susceptibility.
Poirier, L. A.
Abstracts of Papers, American Chemical Society **172**, AGFD 31 (1976) [En] [Nat. Cancer Inst., Nat. Inst. of Health, Bethesda, Maryland 20014, USA]

Dietary alteration can often greatly modify the activities of chemical carcinogens in experimental animals, e.g. severe caloric restriction diminishes the incidence of carcinogen-induced tumour in rodents, riboflavin inhibits the production of liver cancer in rats by N,N-dimethyl-4-aminoazobenzene. The metabolic activation of chemical carcinogens to reactive intermediates in vivo can be significantly altered by naturally-occurring food components. Certain dietary constituents may decrease the formation of the

potentially carcinogenic nitrosamines in foods to which sodium nitrite has been added. Certain dietary components may exert their effects at several stages in the carcinogenic process. Observations in experimental animals are generally made using diets that have been rather drastically altered. Their possible relevance to humans can be seen in the accumulated epidemiological evidence linking various tumours in man with nutritional status. AS

11

A comparison of some chromatographic detectors for the analysis of volatile N-nitrosamines.
Fine, D. H.; Rounbehler, D. P.; Sen, N. P.
Journal of Agricultural and Food Chemistry **24** (5) 980-984 (1976) [10 ref. En] [Thermo Electron Res. Cent., Waltham, Massachusetts 02154, USA]

The analysis of volatile N-nitrosamines by thermal energy analysis (TEA) and gas chromatography (GC) is compared with GC-Coulson electrolytic conductivity detector (GC-CECD), GC-high resolution MS (GC-MS), and TLC. Quantitative chromatograms without interfering peaks were obtained by GC-TEA on crude aqueous distillates and on final methylene chloride extracts following extensive cleanup. For GC-CECD, GC-MS and TLC, data of equal quality were only obtained on the final methylene chloride extract after the extract had been conc. by a factor of 50. Because of the high sensitivity of the GC-TEA, even for dilute extracts, a chromatographic peak corresponding to trace levels of N-nitrosopiperidine was found in several samples. The foods examined were fish meal, cooked bacon fat and cooked lean bacon. AS

12

Analysis of nonionic nonvolatile N-nitroso compounds in foodstuffs.
Fine, D. H.; Ross, R.; Rounbehler, D. P.; Silvergleid, A.; Song, L.
Journal of Agricultural and Food Chemistry **24** (5) 1069-1071 (1976) [6 ref. En] [Thermo Electron Res. Cent., Waltham, Massachusetts 02154, USA]

Simple analytical procedures are described which are capable of quantitatively detecting µg/kg levels of non-ionic non-volatile N-nitroso compounds in complex foodstuffs such as cooked bacon, dried beef, luncheon meat, oily and non-oily fish, and liquors (e.g. vodka). High-pressure liquid chromatography is employed, in combination with the N-nitroso specific Thermal Energy Analyser. AS

13

[Residues in meat and meat products. Report on the conference on biocides and environmental contaminants in meat held 22.3.1974.] Rückstände in Fleisch und Fleischerzeugnissen. Bericht über das Kolloquium von Bioziden und Umweltchemikalien in Fleisch am 22.3.1974. [Conference proceedings]
Germany, Federal Republic of, Deutsche Forschungsgemeinschaft
200pp. ISBN 3-7646-1635-0 (1975) [many ref. De, en] Boppard, Federal Republic of Germany; Harald Boldt Verlag KG

The full text of papers presented at this symposium is given, including the following: General considerations on residues in meat and meat products, by R. Hamm (pp. 11-19); and The nitrosamine problem from the scientists' point of view, by K. Möhler (pp. 192-199, 28 ref.). A further 12 papers are abstracted separately, and are in the author index under **Germany, Federal Republic of,** Deutsche Forschungsgemeinschaft [Meat Symposium]. AJDW

14

[Problems with N-nitrosamines in meat products.] Übersicht zum Problem der N-Nitrosamine in Fleischerzeugnissen. (In 'Rückstände in Fleisch und Fleischerzeugnissen' [see FSTA (1977) **9** 2S164].) [Lecture]
Mirna, A.; Nagata, Y.
pp. 167-185 (1975) [98 ref. De, en] [Inst. für Chemie & Physik, Bundesanstalt für Fleischforschung, 865 Kulmbach-Blaich, Federal Republic of Germany]

Formation of nitrosamines in cured meat products is discussed, with reference to: the curing process; nitrosation reactions involving nitrate or nitrite; analytical methods for nitrosamines; effects of ascorbic acid and sodium ascorbate on nitrosamine formation; and legal aspects. Effects of temp., pH and added nitrate or nitrite concn. on nitrosamine formation are discussed, with reference to tables of literature data. AJDW

15

Study of the pathway of formation of nitrosopyrrolidine in fried bacon.
Hwang, L. S.
Dissertation Abstracts International, B **37** (1) 147 Order No. 76-16407 (1976) [En] [Rutgers Univ., State Univ. of New Jersey, New Brunswick, New Jersey 08903, USA]

In an attempt to establish the origin of nitrosopyrrolidine (NP) and its pathway of formation in fried bacon, bacon was injected with ^{14}C-labelled precursors (proline, putrescine, spermidine, nitrosoproline), fried and then analysed for ^{14}C-NP. ^{14}C-NP was detected only in those samples injected with labelled proline and nitrosoproline. Effects of 0-200 ppm $NaNO_2$ on NP formation were studied. NP was not produced from putrescine or spermidine even at the highest nitrite level. Proline produced increasing amounts of NP with increasing nitrite concn. Effect of time of contact of precursors with nitrite in bacon on NP formation was studied at the 200 ppm $NaNO_2$ level; NP formation increased significantly with contact time. Results of the studies indicated that

proline is the precursor of NP and that nitrosoproline is also involved. Approx. 25% of the NP formed during frying of bacon was lost by vaporization; this may constitute a health hazard to restaurant workers. JA

16

Cured meats.
Unilever Ltd.
British Patent 1 440 183 (1976) [En]

Cured meats, e.g. bacon, are treated with sodium nitrite together with ethoxyquin to reduce the formation of carcinogenic nitrosamines when cooked at high temp. IFT

17

Selective detection of N-nitrosamines by gas chromatography using a modified microelectrolytic conductivity detector in the pyrolytic mode.
Rappard, E. von; Eisenbrand, G.; Preussmann, R.
Journal of Chromatography **124** (2) 247-255 (1976) [16 ref. En] [Inst. of Toxicology & Chem., German Cancer Res. Cent., Heidelberg, Federal Republic of Germany]

Pyrolysis of nitrosamines in a modified Hall microelectrolytic conductivity system is the basis for a highly selective and sensitive detn. method, the response of the detector system to nitrosamines being at least 10^7 times greater than that for n-hexane. The detector response to several operational parameters was studied. For open-chain nitrosamines the detection limit was 50 pg and response was linear up to at least 200 ng. A comparison between reductive detection (pyrolysis in H_2 at 820°C) and pyrolytic detection (550°C) of 5 µl of mixed barley and wheat concentrate fortified with 2 nitrosamines (6 ppb of NDMA and 10 ppb of NDPA), shows that pyrolytic detection gives a clean chromatogram with prominent peaks. The carrier gas and food extracts must be moisture-free, because even traces of water spoil the potassium carbonate scavenger. AL

18

[Nitrite, nitrosamines or botulism?]
Snygg, B. G.
SIK Rapport No. 369, 13pp. (1974) [Sv, en] [Svenska Inst. för Konserveringsforskning, Fack, S-400 21 Göteborg-16, Sweden]

The use of nitrate and nitrite in foods is discussed, with reference to: breakdown products and reaction products of nitrite; the mutagenicity and toxicity of nitrite; functions of nitrate and nitrite in foods (cured colour formation, flavour and aroma development, inhibition of microorganisms); formation of carcinogenic nitrosamines by reaction of nitrite with amines or other N compounds in foods or in the stomach; factors influencing nitrosamine formation (e.g. presence of ascorbic acid); inhibitory effects of nitrite on Clostridium botulinum; and the potential for reduction of the nitrate levels in cured products without increasing the risk of growth and toxin formation by Cl. botulinum. A table is given of literature data for nitrosamine conc. in foods. AJDW

19

Differential pulse polarographic determination of nitrosoproline in raw bacon.
Hasebe, K.; Osteryoung, J.
Abstracts of Papers, American Chemical Society **172**, AGFD 115 (1976) [2 ref. En] [Dep. of Civil Eng., Colorado State Univ., Fort Collins, Colorado 80523, USA]

Nitrosopyrrolidine, which can be formed by nitrosation of pyrrolidine or decarboxylation of nitrosoproline, is a potent carcinogen. The precursor, nitrosoproline, occurs in substantial amounts in bacon. The isolation, clean up, and detn. of nitrosoproline in raw bacon are discussed. The published solvent extraction-freeze drying method [FSTA (1975) **7** 8S1078] and solvent extraction method [Japan Analyst (1975) **24**, 107] have problems with reproducibility, in separation of fats and lipids and in separation of electroactive interferences. Model experiments indicated that separations are improved by using gel permeation chromatography. Elution of samples with ethanol provides good separation of nitrosoproline from lipid materials on Sephadex LH-20. AS

20

Trace analysis of 3-hydroxy-1-nitrosopyrrolidine, a non-volatile N-nitrosamine, by combined gas chromatographic-mass spectrometric method.
Sen, N. P.; Miles, W. F.; Seaman, S.; Lawrence, J. F.
Journal of Chromatography **128** (1) 169-173 (1976) [21 ref. En] [Food Res. Div., Health Protection Branch, Ottawa KLA 012, Canada]

A highly specific gas chromatographic-MS technique is described in which 3-hydroxy-1-nitrosopyrrolidine (HNP) is determined at ng quantities after conversion to a stable volatile derivative, 3-methoxy-1-nitrosopyrrolidine (MNP). This is claimed to be the first successful method reported for analysis of HNP. The methylated ether derivative is easy to prepare, and the product is stable and volatile enough to undergo the vacuum distillation clean-up process. It is hoped that the technique will be useful for trace analysis of NHP in foods, etc. It is thought that traces of HNP may be produced during cooking of bacon or other cured meat products, but due to the lack of suitable analytical methods it has not yet been substantiated. AL

21

Formation of nitrosamines. III. Reaction products of glycocyamine and sodium nitrate.
Yamamoto, M.; Yamada, T.; Tanimura, A.
Journal of the Food Hygienic Society of Japan [Shokuhin Eiseigaku Zasshi] **17** (2) 176-181

(1976) [20 ref. En] [Nat. Inst. of Hygienic Sci., Kamiyoga 1-chome, Setagaya-ku, Tokyo, Japan]

The major reaction product of glycocyamine and sodium nitrite under acidic conditions was identified as carboxymethylnitrosourea. Nitrosocyanamide was not detected. [See FSTA (1975) 7 8C304 for part II.] TM

22

Formation of nitrosamines. IV. Kinetical studies on the carboxymethylnitrosourea formation from glycocyamine and sodium nitrite.
Yamada, T.; Yamamoto, M.; Tanimura, A.
Journal of the Food Hygienic Society of Japan [Shokuhin Eiseigaku Zasshi] 17 (2) 182-186 (1976) [19 ref. En] [Nat. Inst. of Hygienic Sci., Kamiyoga 1-chome, Setagaya-ku, Tokyo, Japan]

The initial rate of carboxymethylnitrosourea formation from glycocyamine and sodium nitrite was studied at pH 2.5 and 37° C. The rate was proportional to the concn. of glycocyamine and sodium nitrite. The effects of thiocyanate, citrate, tartrate, NaCl and ascorbate on the rate were also studied. TM

23

[The state and availability for nitrosamine formation of nitrite in meat products.] [Lecture]
Frouin, A.; Jondeau, D.; Thenot, M.
Proceedings of the European Meeting of Meat Research Workers No. 21, 200-202 (1975) [11 ref. Fr, en, de, ru] [Societe Olida & Caby Associes, 50 Rue Raspail, 92304 Levallois, Perret, France]

On the basis of data for the pH and redox potential of meat products, it is calculated that added nitrite is normally present as NO in meat products. Fixing of this NO by groups present in meat (e.g. SH, OH) is briefly considered; studies were conducted (using the Griess method for detn. of NO) on binding of NO by numerous meat constituents (organic acids, amines, ascorbate, imidazole, cysteine, glutathione, water, glycerol, sucrose). Tables of results are given, and discussed in relation to formation of nitrosamines and Perigo-type inhibitors in meat products. [See FSTA (1977) 9 4S605.] AJDW

24

N-nitrosamines and their precursors in bacon: a review. [Review]
Gray, J. I.
Journal of Milk and Food Technology 39 (10) 686-692 (1976) [59 ref. En] [Dep. of Food Sci., Univ. of Guelph, Guelph, Ontario N1G 2W1, Canada]

Since certain N-nitrosamines are highly carcinogenic, formation and isolation of N-nitrosopyrrolidine from bacon has recently received much attention. This review examines the possible precursors contributing to N-nitrosopyrrolidine formation in bacon, how cooking procedures influence the amount of N-nitrosopyrrolidine formed and the possible effects curing ingredients have on the N-nitrosation reaction. AS

25

[Studies on the potential for reduction of residual nitrite contents in Brühwurst sausages.]
Untersuchungen über Reduzierungsmöglichkeiten des Restnitritgehaltes in Brühwürsten. [Thesis]
Oltmer, V.
110pp. (1975) [58 ref. De, en] Hanover, Federal Republic of Germany; Tierärztliche Hochschule

Studies on minimization of the residual nitrite concn. of Brühwurst cooked smoked sausages are described. Variables studied were: added nitrite concn. (0.4 or 0.6% $NaNO_2$, calculated on the basis of the NaCl content of the product); added ascorbic acid (0 or 0.3%); and added haemoglobin (⩽5%). Effects on residual nitrite concn. ⩽7 days after manufacture were determined; tables of results are given. The initial nitrite concn. of 0.4% gives a 6% reduction in residual nitrite concn., as compared to the higher initial nitrite concn. Addition of ascorbic acid reduces residual nitrite concn. by approx. 11%; added haemoglobin reduces residual nitrite concn. by approx. 3.75% for each 1% added haemoglobin. Addition of ⩽4% haemoglobin has no significant effect on the colour of the sausages. Use of these techniques in combination permits reduction of residual nitrite concn. by ⩽30%. These results are discussed in relation to minimization of the danger of nitrosamine formation in cured meat products. AJDW

26

The effect of antioxidants on the production of volatile nitrosamines during the frying of bacon.
Walters, C. L.; Edwards, M. W.; Elsey, T. S.; Martin, M.
Zeitschrift fuer Lebensmittel-Untersuchung und -Forschung 162 (4) 377-385 (1976) [13 ref. En, de] [British Food Manufacturing Ind. Res. Ass., Leatherhead, Surrey, UK]

In model systems simulating bacon frying considerable variation in the production of N-nitrosopyrrolidine (NOPyr) was observed. The presence of ⩽300 ppm ascorbate or erythorbate (iso-ascorbate) led consistently to a fall in or elimination of NOPyr production. Above this concn. of ascorbate, elimination of NOPyr formation persisted in some instances but in others increased production occurred. With α-tocopherol, a fall in NOPyr production during the simulated frying of bacon was obtained, there being a consistent elimination of NOPyr following addition of 500 ppm. Whilst ascorbyl palmitate had a similar action in reducing or eliminating NOPyr production, no synergistic effect between it and α-tocopherol was noted in an experiment combining the 2 antioxidants. When bacon was fried in fat containing 400 or 800 ppm α-tocopherol, the total production of NOPyr and N-dimethylamine (DMN) in the fried bacon, cooked-out fat and condensate was markedly reduced, but not completely eliminated as in the model systems. Similarly, curing of bacon with a brine containing a mixture of α-tocopherol, ascorbyl palmitate and citric acid led to considerable reductions in NOPyr and DMN

output on frying when the combined concn. of the 2 antioxidants was 800 ppm. AS

27

Occurrence of volatile N-nitrosamines in Japanese salmon roe.
Gadbois, D. F.; Ravesi, E. M.; Lundstrom, R. C.
Fishery Bulletin, National Oceanic and Atmospheric Administration **74** (3) 683-686 (1976) [3 ref. En] [N. E. Utilization Res. Cent., Nat. Marine Fisheries Service, Nat. Oceanic & Atmospheric Administration, PO Box 61, Gloucester, Massachusetts 01930, USA]

Red and pink salmon roe products (sujiko) processed in the USA and exported were sampled in Japan, and analysed for N-nitrosamines by GLC. N-nitrosodimethylamine was found at <5 parts/10^9 in all samples but no other N-nitrosamines were evident. JRR

28

Nitrosopyrrolidine formation in fried bacon.
Hwang, L. S.; Rosen, J. D.
Journal of Agricultural and Food Chemistry **24** (6) 1152-1154 (1976) [20 ref. En] [Dep. of Food Sci., Cook Coll., Rutgers, State Univ. of New Jersey, New Brunswick, New Jersey 08903, USA]

Experiments involving ^{14}C-labelled amines indicated that proline is more likely to be the precursor of nitrosopyrrolidine formation in fried bacon than either spermidine or putrescine. The data are consistent with a mechanism involving formation of nitrosoproline from sodium nitrite and free proline during processing with subsequent decarboxylation of nitrosoproline during frying. AS

29

Update on nitrite, nitrate and nitrosamines. [Lecture]
Greenberg, R. A.
Proceedings of the Meat Industry Research Conference pp. 71-76 (1975) [1 ref. En] [Swift & Co., Oak Brook, Illinois 60521, USA]

Research conducted by the USDA is described, in which samples of 'country style' ham were purchased at 4 locations, analysed for nitrate and nitrite and, after frying in ¼ inch slices for 6 min/side at 340° F, for N-nitrosopyrrolidine. The data show no correlation between NO_3^-, NO_2^- and/or nitrosopyrrolidine content. In commercial plant tests, increasing erythorbate to 1000 ppm and limiting nitrite to 120 ppm has had a measurable effect in reducing nitrosopyrrolidine levels in fried bacon. [See FSTA (1977) 9 5S845.] JRR

30

Determination of N-nitrosoproline at the nanogram level.
Wolfram, J. H.; Feinberg, J. I.; Doerr, R. C.; Fiddler, W.
Journal of Chromatography **132** (1) 37-43 (1977) [14 ref. En] [E. Regional Res. Cent., USDA, Philadelphia, Pennsylvania 19118, USA]

N-nitrosoproline (NPro) has been isolated and identified in uncooked bacon. 2 sensitive detection systems are described for its quantitative detn.: (i) denitrosation followed by derivatization of the amino product, proline, with 7-chloro-4-nitrobenzo-2-oxa-1,3-diazole, the highly fluorescent compound formed the being identified and quantitated by TLC or high-pressure liquid chromatography (HPLC); and (ii) preparation of the volatile methyl ester of the intact nitrosoproline, detection by GLC, and confirmation by GLC-MS. The HPLC fluorescence technique (min. detection level corresponding to 0.5 ng NPro) is approx. 10 × as sensitive as the esterification procedure, but proline, not NPro, is detected, so for food samples free proline would have to be removed and generation of proline prior to derivatization would have to be prevented. [See also FSTA (1975) 7 8S1078.] AL

31

Naturally occurring toxicants in foods and their significance. [Lecture]
Munro, I. C.
Clinical Toxicology **9** (5) 647-663 (1976) [40 ref. En] [Health Protection Branch, Ottawa, Ontario, Canada]

Naturally occurring toxins include mycotoxins (e.g. ergot alkaloid and aflatoxins), allergens, chemical factors (e.g. nitrosamines, heavy metals) and plant toxins, such as those responsible for lathyrism. The groups are discussed with reference to current toxicological understanding of their occurrence and effects. JRR

32

[A gas chromatographic method for the determination of volatile nitrosamines in plant material and soil.] Bestimmung flüchtiger Nitrosamine in pflanzlichem Material und Boden mit Hilfe der Gaschromatographie.
Dressel, J.
Zeitschrift fuer Lebensmittel-Untersuchung und -Forschung **163** (1) 11-13 (1977) [16 ref. De, en] [Landwirtschaftliche Versuchssta., BASF AG, D-6703 Limburgerhof, Federal Republic of Germany]

The method consists in direct steam distillation of 250-500 g comminuted plant material or soil, or, if material (milled grain in particular) foams excessively, steam distillation of diethyl ether extract; and GLC examination of distillate. The reliability of the method was established by recovery of approx. 75% of (i) nitrosodimethylamine and approx. 85% of (ii) nitrosodiethylamine added at 0-8 µg to 500-g samples of wheat or barley, or to 250-g samples of lettuce, carrot or tomato. No detectable quantities of (i) or (ii) were found in the named products themselves. SKK

33

[State of 'nitrite' in meat products.] [Lecture]
Frouin, A.; Thenot, M.
Proceedings of the European Meeting of Meat Research Workers No. 22, F9:1-F9:6 (1976) [40 ref. Fr, en, de, ru] [Service de Recherche de la Societe Olida et Caby Ass., 50 Rue Raspail 92304, Levallois-Perret, France]

Na_2SO_3, in alkaline solution, gives a photometric absorption plot with peaks at 263 nm when the solution is pure, 268 and 274.5 nm when NO is present, 263 and 300 nm when NO_3^- is present, and 263 and 350 nm when NO_2^- is present. Using this reaction on cooked ham and dry sausage extracts, previous theoretical studies are corroborated, showing that 'nitrite' is in the form of nitrogen monoxide (NO) in pork. This result is corroborated by means of Zambelli's reagent. This reagent does not give any characteristic peak with NO_2^- when the initial reaction stage, in acid solution, is suppressed, but it gives a peak with NO in the same conditions. Therefore, it may be calculated that nearly all the NO_2^- is decomposed to NO and fixed under this form in meat products. This suggests that nitrosamines are formed only when high nitrite contents saturate the fixation capacities of NO by meat, or with high temp. which release NO and permit its reoxidation, giving NO_2^-. Therefore, a new approach to the problems of quantitative analysis and toxicity of nitrite is necessary, taking account of these aspects. [See FSTA (1977) **9** 6S988.] AS

34

The distribution of N-nitrosopyrrolidine between lean, fat and vapour in frying bacon. [Lecture]
Mottram, D. S.; Edwards, R. A.; Patterson, R. L. S.
Proceedings of the European Meeting of Meat Research Workers No. 22, F11:1-F11-5 (1976) [10 ref. En, fr, de, ru] [Meat Res. Inst., Langford, Bristol, UK]

When bacon rashers were fried after separation into lean, fat and rind components, N-nitrosopyrrolidine was found almost exclusively in the residual fatty tissue and cooked out fat. The nitrosamine in the residual fried material and cooked out fat only represented a small part of the total nitrosamine produced. The cooking vapours were found to contain 70-80% of the total N-nitrosopyrrolidine formed during frying. The total N-nitrosopyrrolidine yield from the fat was at least fifteen times higher than that from the lean. [See FSTA (1977) **9** 6S988.] AS

35

Nitrosamines in foodstuffs. [Review]
Crosby, N. T.
Residue Reviews 64, 77-135 (1976) [many ref. En] [Dep. of Ind., Lab. of the Gov. Chem., Cornwall House, Stamford Street, London SE1 9NQ, UK]

This review examines: the role of NO_3^- and NO_2^- in meat curing; the fate of added NO_2^-; chemical and biological properties of N-nitrosamines; occurrence and interactions of precursor materials in the environment; analytical methods developed to assess the levels of nitrosamines in foods (isolation, cleanup and concn., identification and detn.); occurrence in foods (fish and meat products); and non-volatile and C-nitroso compounds. Attention is confined mainly to the most recent work. RM

36

Formation in vivo of volatile N-nitrosamines in man after ingestion of cooked bacon and spinach.
Fine, D. H.; Ross, R.; Rounbehler, D. P.; Silvergleid, A.; Song, L.
Nature, UK **265** (5596) 753-755 (1977) [11 ref. En] [Thermo Electron Cancer Res. Cent., 85 First Avenue, Waltham, Massachusetts 02154, USA]

In vivo formation of volatile N-nitrosamines is reported in man after ingestion of a midday meal consisting of bacon, spinach and tomato sandwich and beer. N-nitrosodiethylamine was not found in the food but was formed in vivo after the meal, reaching a max. after the concn. of N-nitrosodimethylamine had reached its max. AL

37

The use of nitrate in the manufacture of Gouda cheese. Lack of evidence of nitrosamine formation.
Goodhead, K.; Gough, T. A.; Webb, K. S.; Stadhouders, J.; Elgersma, R. H. C.
Netherlands Milk and Dairy Journal 30 (3/4) 207-221 (1976) [34 ref. En, nl] [Lab. of the Gov. Chemist, London, UK]

Gouda cheeses, made with the normal amount of added $NaNO_3$ (15 g/100 l. milk), were stored at 13°C and the nitrate and nitrite contents estimated after 24 h, 4 days and at weekly intervals during a 14 wk ripening period. The nitrate content decreased from an average of 56 mg/kg, immediately after preparation, to about 30 mg/kg after 6 wk; thereafter the decrease was much slower. The nitrite content of the cheeses was low, reaching a max. of 1 mg/kg after 2-3 wk and then falling to a steady value of 0.5 mg/kg after 4-5 wk. Additions of 20, 40 or 60 g $NaNO_3$/100 l. milk resulted in higher levels of nitrite (up to 2.5 mg/kg) temporarily occurring in the cheese, but no accumulation of nitrite was observed. These same cheeses were analysed for nitrosamines. The results showed that, under the conditions described, the addition of normal amounts of nitrate during the manufacture of Gouda cheese did not lead to formation of detectable amounts of volatile nitrosamine during ripening. MEG

38

Differential pulse polarographic determination of nitrosamines.
Hasebe, K.; Osteryoung, J.
Abstracts of Papers, American Chemical Society 170, ANAL 68 (1975) [En] [Dep. of Microbiol., Colorado State Univ., Fort Collins, Colorado 80523, USA]

Differential pulse polarography was used to determine N-nitrosopyrrolidine and some of its derivatives at trace levels. Using prior mechanistic studies which suggest important parameters for the electrochemical reduction, the authors determined the effects of solvent composition, supporting electrolyte, and pH, and developed suitable chemical and instrumental conditions for analysis. Detection limits of the order of 7×10^{-8}M are achieved. Application to analysis of meat products for N-nitrosamines is discussed. AS

39

Inhibition of nitrosamine formation in fried bacon by propyl gallate and L-ascorbyl palmitate.
Sen, N. P.; Donaldson, B.; Seaman, S.; Iyengar, J. R.; Miles, W. F.
Abstracts of Papers, American Chemical Society 170, AGFD 55 (1975) [En] [Food Res. Div., Health Protection Branch, Ottawa, Ontario K1A OL2, Canada]

Treatment, just prior to frying, of normal nitrite-cured bacon with 1000 ppm of propyl gallate, piperazine, sodium ascorbate or ascorbyl palmitate markedly reduced the formation of nitrosopyrrolidine during cooking. Propyl gallate, piperazine and ascorbyl palmitate were more effective than sodium ascorbate in this respect. When nitrosproline was added to lard or non-nitrite bacon (bacon prepared without nitrite) and the mixture cooked, formation of nitrosopyrrolidine was demonstrated but the yield (0.04-0.2%) was extremely low. Addition of the above-mentioned additives did not inhibit the formation of nitrosopyrrolidine from nitrosoproline. It was, therefore, concluded that these additives inhibit nitrosopyrrolidine formation during cooking of bacon by interfering with reactions other than the decarboxylation step of nitrosoproline. The possibility of using ascorbyl palmitate in cured bacon is discussed. AS

40

[Problems in the use of nitrite.] Probleme bei Verwendung von Nitrit.
Ingram, M.
Fleischwirtschaft 57 (2) 211-217 (1977) [22 ref. De] [Meat Res. Inst., Langford, Bristol BS18 7DY, UK]

This review deals with problems of NO_2^- use for curing Wiltshire-style bacon, with special reference to the vacuum-packed sliced product. Min. concn. necessary for satisfactory colour and flavour formation and for bacteriological safety are discussed and illustrated with data from previous publications and trials over many yr. Min. initial $NaNO_2$ concn. of ≥ 20 ppm was required for good colour, 50 ppm for bacon flavour formation. The bacteriostatic effect was shown to be enhanced by NaCl, ascorbic acid, low pH, low storage temp., and was dependent on initial level of contamination. Some results suggested the formation of inhibiting compounds ('Perigo factors') during heating of meat + $NaNO_2$. Unpublished results showed 100-1000 µg dimethylamine and trimethylamine/kg bacon (not exceeded at any stage during production and storage) and no dimethyl or any other nitrosamine in raw bacon (limit of detection 1 µg/kg). When pork was enriched with 100 ppm dimethylamine, only 15 µg/kg of dimethyl nitrosamine was found in the bacon after curing. Equimolar concn. of NO_2^- and ascorbic acid reduced nitrosamine formation by >5 fold. No generally valid min. NO_2^- concn. for preserving cured meat products could be suggested. RM

41

The effect of ascorbate reductants on N-nitrosamine formation in a model system resembling bacon fat.
Mottram, D. S.; Patterson, R. L. S.
Journal of the Science of Food and Agriculture 28 (4) 352-354 (1977) [15 ref. En] [Meat Res. Inst., Langford, Bristol BS18 7DY, UK]

The nitrosation of dipropylamine and pyrrolidine was examined in 2-phase systems comprising aqueous buffer and non-polar solvent. Contrary to previous observations in purely aqueous systems [see FSTA (1975) 7 7S931] sodium ascorbate increased amine nitrosation in this 2-phase simulated fat system by 5-25 × compared with ascorbate-free controls. Lipophilic ascorbyl palmitate, however, generally reduced nitrosation. AS

42

Dimethylnitrosamine formation in the gastro-intestinal tract of rats.
Hashimoto, S.; Yokokura, T.; Kawai, Y.; Mutai, M.
Food and Cosmetics Toxicology 14 (6) 553-556 (1976) [15 ref. En] [Yakult Inst. for Microbiological Res., 1796 Yaho, Kunitachi, Tokyo, Japan]

Experiments were carried out to demonstrate the formation of dimethylnitrosamine (DMNA) from dimethylamine (DMA) and nitrate in the digestive tract of rats as a result of the activity of the gastro-intestinal bacteria. All the rats were fed a diet supplemented with 0.1% and 0.4% KNO_3. 5 strains of DMNA-forming bacteria were administered to the rats in the experimental group but not to the control group. DMNA was detected in the contents of the stomach, small intestine and caecum of the rats in the former group but not of the controls. DMNA disappeared more rapidly from the upper part of the small intestine than from the stomach, lower part of the small intestine or caecum. VJG

43

Dietary nitrosamines. [Letter]
Juszkiewicz, T.; Kowalski, B.
Food and Cosmetics Toxicology **14** (6) 653-654 (1976) [5 ref. En] [Dep. of Pharmacology & Toxicol., Vet. Res. Inst., Partyzantow 57, 24-100 Pulawy, Poland]

This letter refers to additional data which it says should be added to a previous review on dietary nitrosamines [see FSTA (1976) **8** 12C585] to make it more complete. VJG

44

[Cancer and nutrition.] Krebs und Ernährung.
Möhler, K.
Umschau in Wissenschaft und Technik **77** (8) 236-240 (1977) [18 ref. De, en] [Inst. für Ernährungswissenschaft, Tech. Univ. München, Weihenstephan, Federal Republic of Germany]

Problems with carcinogenic nitrosamines and their precursors in foods are discussed, with reference to: the relation between structure and carcinogenicity of nitrosamines; pH optima for nitrosamine formation; nitrites, nitrates and nitrosamines in cured meat and fish products; formation of nitrosamines during frying of bacon; factors catalysing and inhibiting nitrosamine formation; nitrosamine formation in the human stomach; the high nitrate concn. in some vegetables; and the health hazard from nitrosamines and their precursors in the diet. AJDW

45

Formation of volatile, hydroxylated, and chlorinated N-nitrosamines during the nitrosation of spermidine 3-hydrochloride.
Hildrum, K. I.; Scanlan, R. A.; Libbey, L. M.
Journal of Agricultural and Food Chemistry **25** (2) 252-255 (1977) [8 ref. En] [Dep. of Food Sci. & Tech., Oregon State Univ., Corvallis, Oregon 97331, USA]

2 hydroxylated dialkyl-N-nitrosamines, 3-butenyl-(3-hydroxypropyl)nitrosamine and 4-hydroxybutyl-(2-propenyl)nitrosamine, were identified as products in the reaction between $NaNO_2$ and spermidine.3HCl at pH 3.5 and 80° C. The compounds were identified by MS and IR spectrometry and by characterization of their trifluoroacetate derivatives. In the presence of chloride ions, chlorinated dialkylnitrosamines were identified as nitrosation products from spermidine. The identification was based on MS. Although other isomers may have been formed, 4-chlorobutyl-(2-propenyl)nitrosamine was the major chlorinated product. AS

46

Factors influencing the rate of formation of volatile N-nitrosamines during the nitrosation of spermidine.
Hildrum, K. I.; Scanlan, R. A.
Journal of Agricultural and Food Chemistry **25** (2) 255-257 (1977) [16 ref. En] [Dep. of Food Sci. & Tech., Oregon State Univ., Corvallis, Oregon 97331, USA]

The influence of time, temp., pH and NaCl concn. on the formation of N-nitrosamines during the nitrosation of spermidine was investigated. Nitrosamines were formed from pH 3.0 to 6.0 and from 25° to 80° C. These observations indicate that nitrosamine formation from spermidine is possible over a wide range of biological and environmental conditions providing that the reactants are present in sufficient quantity. AS

47

Determination of N-nitroso compounds which are not amenable to gas chromatography.
Ross, R. D.; Fine, D. H.; Rounbehler, D. P.; Fan, T.; Silvergleid, A.; Song, L.; Morrison, J.
Abstracts of Papers, American Chemical Society 173, AGFD 56 (1977) [En] [Thermo Electron Cancer Res. Dep., 85 First Avenue, Waltham, Massachusetts 02154, USA]

Detn. of N-nitroso compounds which cannot be gas chromatographed can be accomplished with a thermal energy analyser interfaced to a high-performance liquid chromatograph. The thermal energy analyser is a chemiluminescence detector for uniquely labile nitrosyl moieties such as N-nitrosamines and N-nitrosamides. N-nitroso amino acids have been separated with a strong anion exchange column. The extremely nonvolatile N-nitroso diethanolamine can be chromatographed on a permanently bonded NH_2 column. Progress in applying these chromatographic procedures to detn. of non-gas chromatography amenable compounds at environmental levels in food is described. AS

48

3-hydroxy-N-nitrosopyrrolidine. Isolation from heated 4-hydroxy-N-nitrosoproline.
Lee, J. S.; Bills, D. D.; Scanlan, R. A.; Libbey, L. M.
Journal of Agricultural and Food Chemistry **25** (2) 422-423 (1977) [9 ref. En] [Dep. of Food Sci. & Tech., Oregon State Univ., Corvallis, Oregon 97331, USA]

Formation of 3-hydroxy-N-nitrosopyrrolidine from 4-hydroxy-N-nitrosoproline was demonstrated in a model system simulating conditions under which bacon is fried at a pan temp. of 170° C. Identification of the 3-hydroxy-N-nitrosopyrrolidine was based on coincidence of gas chromatographic retention times and comparison of MS fragmentation patterns with those of the authentic compound synthesized by a separate route. AS

49

An examination of some foodstuffs for the presence of volatile nitrosamines.
Gough, T. A.; McPhail, M. F.; Webb, K. S.; Wood, B. J.; Coleman, R. F.
Journal of the Science of Food and Agriculture 28 (4) 345-351 (1977) [49 ref. En] [Lab. of the Gov. Chem., Cornwall House, Stamford Street, London SE1 9NQ, UK]

Over 500 samples of foodstuffs, including meat, fish and dairy products, vegetables, fruit and soups were examined for the presence of volatile nitrosamines. Cooked complete meals were studied in order to assess the possibility of interactions of the constituents which could result in nitrosamine formation. The extracts of foods were subjected to GLC and the presence of nitrosamines was confirmed using on-line high resolution MS. The most frequently occurring nitrosamines were N-nitrosodimethylamine and N-nitrosopyrrolidine which were present in many cooked bacon samples. N-nitrosodimethylamine also occurred in some other meat products, but other nitrosamines were rarely found. N-nitrosodimethylamine also occurred in fish and cheese, but neither this nitrosamine nor any other was found above the detection limit of 1 μg/kg in any of the other foods. AS

50

Catalytic effect of nitrosophenols on N-nitrosamine formation.
Davies, R.; McWeeny, D. J.
Nature, UK 266 (5603) 657-658 (1977) [7 ref. En] [Food Sci. Div., Min. of Agric. Fisheries & Food, Food Lab. (Colney), Colney Lane, Norwich, UK]

It is reported that nitrosophenols can catalyse N-nitrosamine formation. The effect of (i) p-nitroso-o-cresol on the reaction of 0.15M sodium nitrite with 0.15M pyrrolidine in citrate buffer, pH 5.0, at 37°C was studied at 0.25, 0.50, 0.75 and 1.0 mM concn. using the method of initial rates. The rate of nitrosopyrrolidine (NPy) formation with concn. of (i) was determined. p-nitrosothymol and 1-nitroso-2-napthol also catalysed NPy formation at pH 5.0. No such reaction took place in the absence of nitrite. These observations elucidate the basis for some of the conflicting reports of the effects of phenolic compounds on the rate of nitrosamine formation, but implications for formation of nitrosamines in cured meats and in vivo are not clear. The effects of phenols at $\leq$300 mg/kg, which may be found in smoked meats, and of the 42-300 mg of phenolic material excreted daily into the stomach via gastric juice are likely to be much more complex. AL

51

[Quantitative determination of carcinogenic N-nitrosamines in water by gas chromatography.]
Mikhailovskii, N. Ya.; Rumyantsev, P. G.; Korolev, A. A.; Il'nitskii, A. P.
Gigiena i Sanitariya No. 2, 71-73 (1977) [9 ref Ru] [I Moskovskii Med. Inst. im. I. M. Sechenova, Moscow, USSR]

52

[Role of food factors in the endogenous synthesis of carcinogenic N-nitroso compounds. Review of the literature.] [Review]
Rubenchik, B. L.
Voprosy Pitaniya No. 3, 68-75 (1977) [41 ref. Ru] [Kievskii Nauchno-issled. Inst. Gigieny Pitaniya Ministerstva Zdravookhraneniya USSR, Kiev, USSR]

The subject is discussed under the headings: sources of access of nitrates, nitrites and amines into foods; possible nitrosation of biogenic amines and their derivatives in the body; conditions required for the nitrosation of amines; and is endogeneous nitroso compound synthesis dangerous to health? HBr

53

The absorption and metabolism in rats of small oral doses of dimethylnitrosamine. Implication for the possible hazard of dimethylnitrosamine in human food.
Diaz Gomez, M. I.; Swann, P. F.; Magee, P. N.
Biochemical Journal 164 (3) 497-500 (1977) [29 ref. En] [Courtauld Inst. of Biochem., Middlesex Hospital Med. School, Mortimer Street, London W1P 7PN, UK]

54

[Nitrosamines and their precursors in foods.] [Review]
Miskiewicz, W.
Bromatologia i Chemia Toksykologiczna 10 (1) 1-5 (1977) [31 ref. Pl] [Zaklad Bromatologii Akad. Med., ul. Banach 1, 02-097 Warsaw, Poland]

55

N-nitrosamine precursors in bacon.
Gray, J. I.
Canadian Institute of Food Science and Technology Journal 10 (1) A15-A17 (1977) [14 ref. En] [Dep. of Food Sci., Univ. of Guelph, Guelph, Canada]

The possible precursors of N-nitrosopyrrolidine (NPy) in fried bacon, i.e. proline (and N-nitrosoproline), putrescine, spermidine, and collagen are discussed, together with a possible pathway for free proline, the most probable precursor. The major factors influencing NPy formation, i.e. cooking method, $NaNO_2$ concn., $NaNO_3$ concn., and presence of ascorbic acid, are also discussed. AL

56

Recent nitrosamine analyses in cooked bacon.
Sen, N. P.; Donaldson, B.; Seaman, S.; Collins, B.; Iyengar, J. Y.
Canadian Institute of Food Science and Technology Journal 10 (1) A13-A15 (1977) [18 ref. En] [Canada Dep. of Health & Welfare, Food Res. Div., Ottawa, Ontario, Canada]

The results of a survey in the Ottawa area in

1975 by the Canadian Health Protection Branch are reported and show mean contents of 45 ppm $NaNO_3$ and 62 ppm $NaNO_2$ in uncooked bacon, and 15 parts/billion N-nitrosopyrrolidine (NPy) in cooked lean and 25 parts/billion NPy in cooked fat. A statistical comparison of this data with that obtained in 3 earlier surveys, again in the Ottawa area only, indicates that levels of total NPy (in µg produced by cooking 1 kg raw bacon) were 12, 24, 29 and 13 in 1975, 1974, 1972 and 1970 respectively. AL

57

Nitrites and nitrosamines in perspective.
Rubin, L. J.
Canadian Institute of Food Science and Technology Journal 10 (1) A11-A13 (1977) [8 ref. En] [Canada Packers Ltd., Toronto, Ontario M6N 1K4, Canada]

The problem of nitrites and nitrosamines in the diet, particularly in cured meat products and bacon, is discussed. But such cured products are not the only source of ingested nitrite, they may contribute only up to one third, the rest being formed from ingested nitrate by microflora in the mouth. The nitrate comes from common leafy vegetables (e.g. spinach, lettuce, beets, radish) and even via drinking water. Nitrosamines are not limited to cured meats, but are also found in urban air, soil, water, and sewage-treatment wastes. The question of toxicology is briefly discussed. AL

58

Formation of N-nitrosohydroxypyrrolidine in model and cured meat systems.
Gray, J. I.; Collins, M. E.; Russell, L. F.
Canadian Institute of Food Science and Technology Journal 10 (1) 36-39 (1977) [12 ref. En, fr] [Dep. of Food Sci., Univ. of Guelph, Guelph, Ontario, Canada]

The formation of N-nitrosohydroxypyrrolidine from hydroxyproline and N-nitrosohydroxyproline in model systems was investigated. Decarboxylation of N-nitrosohydroxyproline in an oil-water system produced varying amounts of the N-nitrosamine, depending on the temp. of the system. Methanol was used to extract N-nitrosohydroxypyrrolidine from the systems; a 20% recovery was achieved. Although N-nitrosopyrrolidine was isolated from bacon (contents determined ranging from traces to 41 parts/billion), no N-nitrosohydroxypyrrolidine was detected. Studies with collagen also revealed that little or no N-nitrosohydroxypyrrolidine was formed under the conditions employed. AS

59

A comparison of various mass spectrometric and a chemiluminescent method for the estimation of volatile nitrosamines.
Gough, T. A.; Webb, K. S.; Pringuer, M. A.; Wood, B. J.
Journal of Agricultural and Food Chemistry 25 (3) 663-667 (1977) [25 ref. En] [Dep. of Ind., Lab. of the Gov. Chem., London SE1 9NQ, UK]

Extracts of a variety of commodities (including canned luncheon meat, bacon, vegetation and Chinese salted fish, sauce and soybean foods) have been analysed for the presence of volatile nitrosamines using high- and low-resolution MS and chemiluminescent detection. The quantitative results obtained by these methods have been compared to assess their reliability. The high-resolution peak matching and chemiluminescent data are in agreement with each other for all the analyses, but inconsistencies were observed using the other MS procedures. AS

60

N-nitroso compounds in the environment.
[Conference proceedings]
Bogovski, P.; Walker, E. A.; Davis, W.
International Agency for Research on Cancer; World Health Organization (Editors)
IARC Scientific Publications No. 9, xv + 243pp. (1974) [many ref. En] 1211 Geneva 27, Switzerland; World Health Organization Price SF50.00 [IARC, Lyon, France]

The proceedings of a working conference held at the International Agency for Research on Cancer, Lyon, France, on 17-20 Oct. 1973 contained the following sessions: Analysis of N-nitroso compounds (pp. 1-63); Formation of N-nitroso compounds (pp. 65-144); and N-nitroso compounds in the environment (pp. 145-236). Individual papers include: Organisational and research activities on the analysis of N-nitroso compounds at the International Agency for Research on Cancer, by P. Bogovski (pp. 3-5); Recent developments in trace analysis of volatile nitrosamines: a brief review, by G. Eisenbrand (pp. 6-11, 46 ref.); Progress in the analysis of volatile N-nitroso compounds, by G. M. Telling, T. A. Bryce, D. Hoar, D. Osborne & D. Welti (pp. 12-17, 6 ref.); Analysis of volatile nitrosamines: progress in gas chromatography-mass spectrometry techniques, by T. A. Gough & R. Sawyer (pp. 18-21, 6 ref.); The determination of total non-volatile nitrosamines in microgram amounts, by C. L. Walters, D. G. Fueggle & T. G. Lunt (pp. 22-25, 7 ref.); Nitrosamine analysis, II: the isolation and chromatography of some high molecular weight N-nitroso compounds, by F. T. Kelly & J. R. Nunn (pp. 26-31, 8 ref.); A solvent-partitioning, high-speed, liquid chromatographic procedure for clean-up in the analysis of nitrosamines, by W. T. Iwaoka, M. S. Weisman & S. R. Tannenbaum (pp. 32-35, 9 ref.); [continued in following abstr.] AL

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N-nitroso compounds in the environment.
[Conference proceedings]
Bogovski, P.; Walker, E. A.; Davis, W.
International Agency for Research on Cancer; World Health Organization (Editors)
IARC Scientific Publications No. 9, xv + 243pp. (1974) [many ref. En] 1211 Geneva 27, Switzerland; World Health Organization Price

SF50.00 [IARC, Lyon, France]

[Continued from preceding abstr.] Some remarks on the identification and quantitative determination of nitrosamines, by R. Truhaut (pp. 36-39, 22 ref.); Description of the thermal energy analyser for N-nitroso compounds, by D. H. Fine & F. Rufeh (pp. 40-44, 11 ref.); An investigation of the possible value of oxonium salt formation in nitrosamine analysis, by M. Castegnaro, B. Pignatelli & E. A. Walker (pp. 45-48, 5 ref.); The analysis and occurrence of volatile nitrosamines in cured meat products, by N. P. Sen, J. R. Iyengar, B. Donaldson, T. Panalaks & W. F. Miles (pp. 49-52, 13 ref.); Analysis of μg/kg levels of non-volatile and volatile N-nitroso compounds in foodstuffs, by D. H. Fine & F. Rufeh (pp. 53-56, 2 ref.); A report on the present status of a collaborative study of methods for the trace analysis of volatile nitrosamines, by E. A. Walker & M. Castegnaro (pp. 57-62); Formation of N-nitroso compounds, preparation of heptafluorobutyryl derivatives of ureas, and fate of nitrite in the rat stomach, by S. S. Mirvish, S. Arnold, E. Conrad, P. Ghadirian, V. R. C. Kommineni & J. Sams (pp. 67-70, 12 ref.); Formation of N-nitroso compounds from agricultural chemicals and nitrite, by G. Eisenbrand, O. Ungerer & R. Preussmann (pp. 71-74, 11 ref.); Formation of nitrosodimethylamine from the interaction of certain pesticides and nitrite, by N. P. Sen, B. Donaldson & C. Charbonneau (pp. 75-79, 26 ref.); [continued in following abstr.] AL

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N-nitroso compounds in the environment.
[Conference proceedings]
Bogovski, P.; Walker, E. A.; Davis, W.
International Agency for Research on Cancer; World Health Organization (Editors)
IARC Scientific Publications No. 9, xv + 243pp. (1974) [many ref. En] 1211 Geneva 27, Switzerland; World Health Organization Price SF50.00 [IARC, Lyon, France]

[Continued from preceding abstr.] Catalysis of nitrosamine synthesis, by F. Schweinsberg (pp. 80-85, 18 ref.); Catalysis of nitrosation reactions by electrophilic species, by P. P. Roller & L. K. Keefer (pp. 86-89, 15 ref.); The nitrosation of spermidine and spermine, by J. H. Ferguson, T. J. Mysliwy & M. C. Archer (pp. 90-93, 8 ref.); Transnitrosation and decomposition of nitrosamines, by A. J. Buglass, B. C. Challis & M. R. Osborne (pp. 94-100, 22 ref.); Prevention by ascorbic acid of in vivo formation of N-nitroso compounds, by S. Ivankovic, R. Preussmann, D. Schmähl & J. W. Zeller (pp. 101-102, 11 ref.); The effect of ascorbic acid and glutathione on the formation of nitrosopiperazines from piperazine adipate and nitrite, by N. P. Sen & B. Donaldson (pp. 103-106, 10 ref.); The interaction of nitrite with α-amino-acid and peptide derivatives: the ^{15}N-nuclear magnetic resonance approach, by R. Bonnett & R. Holleyhead (pp. 107-110, 15 ref.); Formation of nitrosamines from tertiary amines and nitrous acid, by W. Lijinsky & G. M. Singer (pp. 111-114, 9 ref.); The potential formation of C-nitroso compounds in smoked, cured meats, by M. E. Knowles, J. Gilbert & D. J. McWeeny (pp. 115-120, 23 ref.); Experimental production of nitrosamines in vivo, by V. Galea, N. Preda, L. Popa & G. Simu (pp. 121-122, 9 ref.); Quantitative measurement of in vivo nitrosamine formation, by J. Sander, J. LaBar, M. Ladenstein & F. Schweinsberg (pp. 123-131, 2 ref.); [continued in following abstr.] AL

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N-nitroso compounds in the environment.
[Conference proceedings]
Bogovski, P.; Walker, E. A.; Davis, W.
International Agency for Research on Cancer; World Health Organization (Editors)
IARC Scientific Publications No, 9, xv + 243pp. (1974) [many ref. En] 1211 Geneva 27, Switzerland; World Health Organization Price SF50.00 [IARC, Lyon, France]

[Continued from preceding abstr.] Thiocyanate catalysis of nitrosamine formation and some dietary implications, by E. Boyland & S. A. Walker (pp. 132-136, 13 ref.); N-nitrosation of secondary amines, and particularly of drugs, in buffer solutions and human gastric juice, by D. Ziebarth (pp. 137-141, 25 ref.); Possible mechanisms of nitrosamine formation in food and animal feeding stuffs, by K. Möhler, O. L. Mayrhofer & E. Hallermayer (pp. 142-143, 8 ref.); Nitrosamines in cured meat products, by A. E. Wasserman & W. Fiddler (pp. 147-150, 20 ref.); Recent studies in Japan on the analysis and formation of N-nitroso compounds, by K. Kojima & M. Fujita (pp. 151-153, 13 ref.), with an addendum by T. Kawabata (pp. 154-158, 3 ref.); Gas chromatographic trace analysis of volatile nitrosamines in various types of wheat flour after application of different nitrogen fertilisers to the wheat, by K. Heyns & H. Röper (pp. 166-172, 10 ref.); Passage of nitrosamines from rumen into milk in goats, by T. Juszkiewicz & B. Kowalski (pp. 173-176, 6 ref.); Determination of nitrous gases in smoke, by J. Kann, O. Tauts, R. Kalve & T. Paalme (pp. 180-182, 13 ref.); Determination of volatile N-nitroso compounds, and particularly of nitrosodimethylamine, nitrosodiethylamine and nitrosodipropylamine, in soyabean oil: effect of oil storage period on recovery rates, by L. Hedler & P. Marquardt (pp. 183-191, 4 ref.); [continued in following abstr.] AL

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N-nitroso compounds in the environment.
[Conference proceedings]
Bogovski, P.; Walker, E. A.; Davis, W.
International Agency for Research on Cancer; World Health Organization (Editors)
IARC Scientific Publications No. 9, xv + 243pp.

(1974) [many ref. En] 1211 Geneva 27, Switzerland; World Health Organization Price SF50.00 [TARC, Lyon, France]

[Continued from preceding abstr.] Some evidence of the presence of traces of nitrosamines in cider distillates, by P. Bogovski, E. A. Walker, M. Castegnaro & B. Pignatelli (pp. 192-196, 17 ref.); The effect of ascorbate on amine-nitrite hepatoxicity, by J. J. Kamm, T. Dashman, A. H. Conney & J. J. Burns (pp. 200-204, 13 ref.); Experiments on the degradation of nitrosamines by plants, by J. Sander, M. Ladenstein, J. LaBar & F. Schweinsberg (pp. 205-210, 7 ref.); Investigations on amines in the human environment, by G. B. Neurath & O. Schreiber (pp. 211-214, 13 ref.); Some studies on the production of nitrosamines in the urinary bladder and their subsequent effects, by M. J. Hill & G. Hawksworth (pp. 220-222, 4 ref.); The precursors of N-nitroso compounds in foods, by C. L. Walters, B. E. Newton, D. V. Parke & R. Walker (pp. 223-228, 10 ref.); and Possible relationship between nitrates, nitrosamines and gastric cancer in south-west Colombia, by G. Hawksworth, M. J. Hill, G. Gordillo & C. Cuello (pp. 229-234, 7 ref.). Reports and recommendations (pp. 239-243) are included. AL

65

[Toxicological evaluation of nitrosamine precursors.] Die gesundheitliche Beurteilung von Nitrosaminbildnern. [Lecture]
Marquardt, P.
Qualitas Plantarum - Plant Foods for Human Nutrition 27 (1) 85-98 (1977) [17 ref. De, en] [Laufenerstrasse 9, 7800 Freiburg/Brsg., Federal Republic of Germany]

Aspects considered in this discussion of nitrosamines include: structure and carcinogenicity; organ-specificity of carcinogenic activity of nitrosamines; precursors of nitrosamines (nitrates, nitrites, oxides of nitrogen, and primary or secondary amines); factors influencing nitrosamine formation (pH, relative concn. of precursors, presence of catalysts or inhibitors); nitrates in drinking water and foods of vegetable origin; bacterial reduction of nitrate to nitrite; nitrosamines in cured meat products; and problems of establishment of an acceptable daily intake value for nitrosamines. [See FSTA (1977) 9 11A722.] AJDW

66

Survey of finfish and shellfish for volatile N-nitrosamines.
Havery, D. C.; Fazio, T.
Journal of the Association of Official Analytical Chemists 60 (3) 517-519 (1977) [15 ref. En] [Div. of Chem. & Physics, FDA, Washington, DC 20204, USA]

A variety of finfish and shellfish obtained on the local retail market were analysed for 14 volatile N-nitrosamines by using a GLC method demonstrated to be sensitive to 10 µg/kg. A total of 78 samples (fresh, frozen and canned), representing 26 varieties of products, were analysed. No nitrosamines were found. AS

67

Effect of irradiation on the content of nitrosamines in meat and meat products.
Klein, S.; Pipek, P.; Velisek, J.
Sbornik Vysoke Skoly Chemicko-Technologicke v Praze, E No. 48, 33-41 (1976) [21 ref. En, cs, ru] [Fac. of Food & Biochem. Tech., Prague Inst. of Chem. Tech., Prague, Czechoslovakia]

The effect was investigated of ionizing radiation on nitrosamine formation in meat. Samples of beef muscle homogenates containing different amounts of sodium nitrite (0, 10 and 100 mg/100 g) were irradiated with different doses of γ-rays (0, 5, 10, 50 kJ/kg). Sodium nitrite was added either before or after irradiation. In no case did the level of dimethylnitrosamine detected by GLC exceed 80 µg/kg. AS

68

[Nitrite fixation by muscle proteins.] [Lecture]
Rougie, P.; Valin, C.; Goutefongea, R.
Annales de la Nutrition et de l'Alimentation 30 (5/6) 759-765 (1976) [many ref. Fr, en] [Sta. de Recherche sur la Viande, INRA, Theix, 63110 Beaumont, France]

The toxicity of NO_2^- and especially of nitrosamines is reviewed in relation to the presence of free or bound NO_2^- in meat products. The binding of NO_2^- in cured meats is discussed, together with methods of analysis. [See FSTA (1977) 9 11A688.] RM

69

[State of nitrite in meat products.] [Lecture]
Frouin, A.; Thenot, M.; Jondeau, D.; Barraud, C.; Grimault, M.-L.; Durand, P.; Vendeuvre, J. L.
Annales de la Nutrition et de l'Alimentation 30 (5/6) 773-788 (1976) [42 ref. Fr, en] [Soc. Olida-Caby, 50 Rue de Raspail, 92300 Levallois-Perret, France]

The fate of NO_2^- in cured meat products was investigated. Analysis of many samples of ham and dry sausages by a variety of methods provided no evidence for its oxidation to NO_3^-. Theoretical calculations based on the physico-chemical conditions prevailing in cured meat products suggested a total breakdown of NO_2^- to NO, which can be bound by many of the meat components. Results of the calculations were confirmed by partial vacuum distillation (under 5 cm Hg pressure), absorption spectra in alkaline sulphite medium [see FSTA (1974) 6 7S890] and the modified Zambelli reaction. All the technological and toxicological effects attributed to NO_2^- are

thus explained in terms of more or less strongly bound NO, depending on the medium. Accordingly, nitrosamine would be formed either at very high NO_2^- addition or at high temp., and the risk of its formation can be further reduced by addition of NO-binding compounds, e.g. ascorbic acid. [See FSTA (1977) 9 11A688.] RM

70

Nitrosation of food amines under stomach conditions. (In 'Proceedings of the Second International Symposium on Nitrite in Meat Products' [see FSTA (1977) 9 12S2047].) [Lecture]
Walters, C. L.
pp. 125-133 (1977) [En] [British Food Manufacturing Ind. Res. Ass., Leatherhead, Surrey, UK]

Studies on nitrosation of food amines under the conditions occurring in the human stomach are discussed; both in-vivo and in-vitro trials were conducted. Aspects considered included: nitrite concn. in saliva (with special reference to effects of consumption of nitrate-rich foods, e.g. lettuce); effects of diet on the nitrite concn. in the stomach contents; the concn. of thiocyanate (a nitrosation-stimulating agent) in the stomach contents of smokers and non-smokers; acid secretion in the stomach in relation to endogenous nitrite and thiocyanate concn.; and in-vivo studies on nitrosamine formation in the stomach of people consuming a diet containing 0.46-0.77 mmol nitrite/l. Traces of nitrosopiperidine were detected in stomach contents samples from some volunteers. AJDW

71

A new type of N-nitrosation inhibitor. (In 'Proceedings of the Second International Symposium on Nitrite in Meat Products' [see FSTA (1977) 9 12S2047].) [Lecture]
Groenen, P. J.
pp. 171-172 (1977) [En] [Cent. Inst. for Nutr. & Food Res. TNO, Utrechtseweg 48, Zeist, Netherlands]

A brief account is given of recent identification of pyrrole as a nitrosation inhibitor in tobacco; the inhibitory activity of pyrrole is at least equal to that of 4-methyl catechol, which is known to be a highly effective nitrosation inhibitor. The possible implications of this observation for nitrosation reactions in foods is discussed, with reference to the common occurrence of pyrrole and related compounds in foods. AJDW

72

Developments in nitrosamine analysis. (In 'Proceedings of the Second International Symposium on Nitrite in Meat Products' [see FSTA (1977) 9 12S2047].) [Lecture]
Castegnaro, M.; Walker, E. A.
pp. 187-190 (1977) [En] [Int. Agency for Res. on Cancer, 150 Cours Albert Thomas, 69008 Lyon, France]

A brief review is given of developments in methods for analysis of nitrosamines. The screening method for volatile nitrosamines used by the International Agency for Research on Cancer is described [see FSTA (1976) 8 4A188], with special reference to a recent modification to the method for oxidation of nitrosamines to nitramines. The original method was based on oxidation with 50% H_2O_2 in water in the presence of trifluoroacetic acid; this gives a nitramine yield of approx. 50%. Telling [see FSTA (1973) 5 2C42] achieved a yield of 85% with an 85% solution of H_2O_2. This highly conc. H_2O_2 is, however, hazardous and difficult to obtain. The modified method described uses 50% H_2O_2 in the presence of the extraction solvents; a table of results shows that yield is similar to that achieved by Telling's technique. Introduction of MS for confirmatory testing of nitramines is also discussed. 3 collaborative studies on methods for detn. of volatile nitrosamines are described. Both within and between laboratories, agreement is less precise at 5 µg/kg than at 20 µg/kg. MS gives more precise results than GLC at low nitrosamine concn., especially for nitrosodimethylamine. The potential for use of the thermal energy analyser is also considered. AJDW

73

Trace analysis of polar and apolar N-nitroso compounds by combined high-performance liquid chromatography and thermal energy analysis. (In 'Proceedings of the Second International Symposium on Nitrite in Meat Products' [see FSTA (1977) 9 12S2047].) [Lecture]
Fine, D. H.; Rounbehler, D. P.; Silvergleid, A.; Ross, R.
pp. 191-198 (1977) [En] [Thermo Electron Res. Cent., 85 First Avenue, Waltham, Maryland 02154, USA]

Thermal energy analysis (TEA) has been combined with high-pressure liquid chromatography (HPLC). Performance characteristics, including sensitivity, linearity, reproducibility, selectivity and proportionality of response to amount of substance, are discussed. The most useful feature of TEA-HPLC is unique selectivity to the nitrosyl functional group, allowing detection of mass fractions $< 10^{-9}$ or mass concn. < 1 µg/l., even in the presence of 'crude' environmental samples. TEA-HPLC allows, for the first time, routine analysis of non-volatile N-nitroso compounds in the environment. AS

74

Some critical remarks on the optimum resolution to use in trace analysis of nitrosamines with combined gas chromatography and mass spectrometry. (In 'Proceedings of the Second International Symposium on Nitrite in Meat Products' [see FSTA (1977) 9 12S2047].) [Lecture]
Stephany, R. W.
pp. 239-248 (1977) [En] [Nat. Inst. of Public Health (RIV), Antonie van Leeuwenhoeklaan 9, PO Box 1, Bilthoven, Netherlands]

The resolution of the mass spectrometer, the quality of its ion optics together with the properties of the gas chromatographic column are discussed as critical parameters in the analysis of N-nitrosamines with the combination GLC-MS. AS

75

How specific and sensitive is the thermal energy analyser? (In 'Proceedings of the Second International Symposium on Nitrite in Meat Products' [see FSTA (1977) 9 12S2047].) [Lecture]
Stephany, R. W.; Schuller, P. L.
pp. 249-255 (1977) [En] [Nat. Inst. of Public Health (RIV), Antonie van Leeuwenhoeklaan 9, PO Box 1, Bilthoven, Netherlands]

Various aliphatic, alicyclic and aromatic C-nitroso compounds are reported to give a weak to strong molar response on the thermal energy analyser (TEA), depending on the structure of the compound considered. As a consequence, these compounds may interfere and give false positive results in the analysis of N-nitrosamines with TEA and GLC-TEA. AS

76

[Amines and nitrosamines. Occurrence, significance, metabolism and determination.] Amine und Nitrosamine. Vorkommen, Bedeutung, Stoffwechsel und Bestimmung.
Askar, A.
Fortschritte in der Lebensmittelwissenschaft No. 4, vi + 222pp. ISBN 3-7983-0204-9 (1976) [many ref. De] Berlin; Universitätsbibliothek der Technischen Universität [Inst. für Lebensmitteltech., Frucht- & Gemüsetech., Tech. Univ., Königin-Luise-Strasse 22, 1 Berlin (West) 33]

This book, the fourth in the series 'Fortschritte in der Lebensmittelwissenschaft' includes the following chapters: Biosynthesis of amines (pp. 5-13, 13 ref.); Occurrence of amines in various foods [milk, cheese, fish, wine, beer, sauerkraut, fruit, vegetables, seeds, fungi, tea, yeast extract and walnuts] (pp. 14-72, many ref.); The significance of amines in foods (with reference to their importance as aroma substances, in non-enzymic browning, as precursors of alkaloids, as a quality control criterion, as a source of poisoning, and to the functions of di- and polyamines, functions of biogenic amines and the use of biogenic amines in diagnosis] (pp. 73-103, many ref.); Metabolism of amines (pp. 104-139, 25 ref.); Nitrosamines (pp. 140-151, 33 ref.); Determination of amines [covering extraction, concentration, analysis by paper chromatography, TLC, GLC, MS, column chromatography and fluorometric methods, detn. of trimethylamine in fish, and detn. of nitrosamines] (pp. 152-199, many ref.); and Summary (pp. 203-213). A 7-pp. subject index is included. AJDW

77

Nitrates and nitrites: reinterpretation of analytical data by means of bound nitrous oxide. (In 'Proceedings of the Second International Symposium on Nitrite in Meat Products' [see FSTA (1977) 9 12S2047].) [Lecture]
Frouin, A.
pp. 115-120 (1977) [En] [Res. Dep. of the Olida-Caby Cie, 50 Rue Raspail, 92304, Levallois-Perret, France]

Discrepancies in detn. of nitrate and nitrite in cured meat products by various methods (including French Standard methods NF-V4409 and 4410) are discussed, with special reference to the state of added nitrite in meat products. Theoretical considerations and further analyses suggest that the 'nitrite' present in cured meats is reduced to NO; this is then bound to proteins, thiols, carbonyl compounds, etc. in the meat. There is, therefore, a complex equilibrium between the various NO-binding constituents; analytical reagents upset this equilibrium, thereby releasing variable amounts of NO. This hypothesis of formation and binding of NO is discussed in relation to nitrosamine formation in cured meats. AJDW

78

Formation of carcinogenic nitrosamines in relation to nitrite in meat products. (In 'Proceedings of the Second International Symposium on Nitrite in Meat Products' [see FSTA (1977) 9 12S2047].) [Lecture]
Sander, J.; Fetterroll, B.
pp. 137-144 (1977) [En] [Staatliches Medizinaluntersuchungsamt, Alte Poststrasse 11, 45 Osnabrück, Federal Republic of Germany]

Factors related to the possible hazard of formation of nitrosamines from nitrite and amines in the human stomach were studied. Results showed that: nitrite ingested in ham broth disappeared from the stomach after 20-30 min, while nitrite ingested in boiled ham remained in the stomach for longer; the amine amidopyrine disappeared from the stomach approx. 10 min after ingestion; appreciable in-vitro formation of nitrosodimethylamine was observed as a result of reaction of amidopyrine with nitrite present in spinach or cured meats; and that no appreciable formation of nitrosamines from nitrosated amino acids by bacterial decarboxylation occurred in in-vitro studies. AJDW

79

Analysis, formation and occurrence of volatile and non-volatile N-nitroso compounds: recent results. (In 'Proceedings of the Second International Symposium on Nitrite in Meat Products' [see FSTA (1977) 9 12S2047].) [Lecture]
Eisenbrand, G.; Janzowski, C.; Preussmann, R.
pp. 155-169 (1977) [En] [Inst. for Toxicology & Chemotherapy, German Cancer Res. Cent., D6900 Heidelberg, Federal Republic of Germany]

In 17 out of 52 cured meat products, volatile nitrosamines (1-66 μg/kg) were detected. After

frying, all of the products tested (18) contained nitrosamines. Contents of N-nitrosopyrrolidine and N-nitrosopiperidine were substantially higher in most samples after the frying process. A method for the detection of N-nitroso-3-hydroxypyrrolidine [by GLC with a thermal energy analyser (TEA)] at levels of μg/kg in foods was developed. In 7 out of 11 different cured meat products, the compound was detected at contents of ⩽9 μg/kg after frying. Heat-induced decarboxylation of N-nitrososarcosine, N-nitrosoproline and N-nitroso-3-hydroxyproline was studied in model systems and the relationship between heating temp. and yield of nitrosamines investigated. A method of trace analysis for nitrosamino acids in foods was developed [based on GLC with TEA detection]. AS

80

Nitrosopyrrolidine in United States cured meat products. (In 'Proceedings of the Second International Symposium on Nitrite in Meat Products' [see FSTA (1977) 9 12S2047].)
[Lecture]
Greenberg, R. A.
pp. 203-210 (1977) [En] [Swift & Co., Oak Brook, Illinois, USA]

Studies on the occurrence and concn. of nitrosopyrrolidine (NP) in cured meat products in the USA are discussed. Tables of data are given showing: effects of added erythorbate and reduced nitrite concn. on NP concn. in fried bacon; trends in NP concn. in fried bacon in the USA during the period 1971-1976; the relation between residual nitrite content and NP formation; and NP concn. in fried country-style ham. The results show that: NP is commonly present in fried bacon, and occasionally present in fried country-style ham; average NP concn. of fried commercial bacon has decreased from 67 to 17 μg/kg over the period 1972-1976; effects of added erythorbate on NP formation were relatively small; and NP content of the fried product was not significantly related to the residual nitrate or nitrite concn. AJDW

81

Volatile nitrosamines in commercial Swiss meat products. (In 'Proceedings of the Second International Symposium on Nitrite in Meat Products' [see FSTA (1977) 9 12S2047].)
[Lecture]
Hauser, E.
pp. 215-219 (1977) [En] [Swiss Fed. Vet. Office, Berne, Switzerland]

In 112 samples of Swiss meat products, including scalded and fermented sausage, cooked cured ham and dried meat, combined GLC-MS failed to demonstrate dimethylnitrosamine, methylethylnitrosamine, diethylnitrosamine, nitrosopyrrolidine, nitrosopiperidine and nitrosomorpholine. The detection limit was a mass fraction of about 1 μg/kg. AS

82

Development of a method of estimating N-nitrosamino acids and its use on some meat products. (In 'Proceedings of the Second International Symposium on Nitrite in Meat Products' [see FSTA (1977) 9 12S2047].)
[Lecture]
Dhont, J. H.
pp. 221-225 (1977) [En] [Cent. Inst. for Nutr. & Food Res. TNO, Utrechtseweg 48, Zeist, Netherlands]

A procedure for detn. of the N-nitrosamino acids nitrosoproline and nitrososarcosine is described, based on extraction with a methanol/water (3:1) mixture, precipitation of impurities by addition of $ZnSO_4$ + NaOH, elimination of methanol from the mixture by evaporation, acidification of the aqueous residue, re-extraction with diethyl ether, addition of water to the extract, evaporation-off of the ether, filtration of the aqueous residue through an Amberlite XAD-2 column then through an acidified cation exchange column, and quantitative analysis by the photolytic nitrite method, polarography or gas chromatography. Recovery was 90-100% in model systems, 80-100% in meat products. This procedure did not give quantitative extraction of nitrosohydroxyproline. Data are given for total nitrosamino acid concn. determined by this method in samples of smoked horse meat, raw and fried bacon, luncheon meat, liver sausage, cooked liver, pate de foie, canned liver pie, Guelder-type ring sausage and raw and fried minced meat. The highest observed concn. of nitrosamino acids was 2.9 mg/kg in canned liver pie. AJDW

83

Formation of nitrosamines in fermented meat products. (In 'Proceedings of the Second International Symposium on Nitrite in Meat Products' [see FSTA (1977) 9 12S2047].)
[Lecture]
Kotter, L.; Schmidt, H.; Fischer, A.
pp. 83-89 (1977) [En] [Inst. for Hygiene & Tech. of Foods of Anim. Origin, Univ. of Munich, Munich, Federal Republic of Germany]

Factors influencing formation of nitrosamines in meat products are discussed on the basis of literature data. Aspects considered include: the need for presence of nitrite and nitrosatable substances e.g. amines; effects of heating time and temp. (with special reference to frying of bacon); nitrosamine concn. in the lean and fat fractions of cured meats; effects of spices, reducing agents, antioxidants, and glucono-δ-lactone on nitrosamine formation in meat products; the role of microorganisms in nitrosamine formation; nitrosatable amines and their precursors in meat; and effects of blood and haemoglobin on nitrosamine formation. 3 possible methods for restriction of nitrosamine formation in foods or in the human stomach are suggested: ensuring that the diet is low in nitrosating agents; ensuring that the diet is low in amines, and that use of amine drugs is

controlled; and excluding handling and processing methods favouring nitrosation reactions. AJDW

84

Irradiation as a conceivable way of reducing nitrites and nitrates in cured meats. (In 'Proceedings of the Second International Symposium on Nitrite in Meat Products' [see FSTA (1977) 9 12S2047].) [Lecture]
Wierbicki, E.; Heiligman, F.; Wasserman, A. E.
pp. 75-81 (1977) [En] [Food Eng. Lab., US Army Natick R&D Command, Natick, Massachusetts, USA]

Studies on the potential use of irradiation to minimize the quantity of nitrite used in cured meats (ham, corned beef, bacon) are described. Data are given for the composition and organoleptic properties of samples cured conventionally, and for irradiated products cured at low nitrite concn. The results show that, in irradiated samples, added nitrite concn. can be reduced to the level required for formation of 'cured' flavour and colour (approx. 25 mg/kg). Small quantities of nitrate are also required, to prevent fading of the cured colour, to supplement the nitrite, and to scavenge electrons formed during irradiation. Total added nitrate + nitrite should be 75 mg/kg (of which 33-50% should be nitrite) in irradiated ham or corned beef, and 50 mg/kg (of which 50% should be nitrite) in bacon. No nitrosamines were detected in irradiated/low nitrite cured ham or pre-fried bacon; low concn. of nitrosopyrrolidine were, however, detected in fried samples of non-precooked irradiated/low nitrite cured bacon. AJDW

85

Processing and sensory evaluation of a primal cut cured with nitrite alone or nitrite-nitrate mixture. (In 'Proceedings of the Second International Symposium on nitrite in meat products' [see FSTA (1977) 9 12S2047].) [Lecture]
Wasserman, A. E.; Fiddler, R. N.
pp. 67-72 (1977) [En] [USDA, Philadelphia, Pennsylvania 19118, USA]

Comparative studies were conducted on curing of beef sirloin tips in (i) normal-type brines containing 7750 mg $NaNO_3$/l. + 1670 mg $NaNO_2$/l., or (ii) experimental brines containing 2200 mg $NaNO_2$/l. The meat was cured for 6 wk, then dried at 65°C for approx. 3 days. The meat was analysed for NaCl, $NaNO_3$, $NaNO_2$ and nitrosamines; microbiological quality of the meat and the curing brine was evaluated, and the meat was evaluated organoleptically. Curing brine composition had little effect on the microflora or NaCl content of the beef. Nitrite and nitrate concn. in the cured dried products were, respectively (mg/kg): (i) 37 and 3097; and (ii) 26 and 103. No nitrosamines were detected in samples cured with (i); dimethylnitrosamine was detected (at a concn. of < 14 µg/kg) in samples cured with (ii). Taste panel trials showed the product cured in (i) to be preferred; both products were, however, well accepted if served as chipped beef in white sauce. AJDW

86

N-nitroso compounds in products widely consumed in France: hams. (In 'Proceedings of the Second International Symposium on Nitrite in Meat Products' [see FSTA (1977) 9 12S2047].) [Lecture]
Klein, D.; Poullain, B.; Debry, G.
pp. 289-292 (1977) [En] [Dep. de Nutr. de des Maladies Metaboliques, Univ. de Nancy I, INSERM U 59, 40 Rue Lionnois, 54000 Nancy, France]

9 samples of ham of various types (cooked, smoked, Bayonne, Westphalian, Parma) were studied. Values for residual $NaNO_2$ contents ranged from 4 to 13 mg/kg; $NaNO_3$ contents ranged from 50 to 363 mg/kg. All 9 samples contained dimethylnitrosamine; concn. observed were (µg/kg): < 1 in 3 samples, 2.0, 2.46, 3.2, 4.2, 9, and 18.4. Presence of dimethylnitrosamine in the sample containing 18.4 µg/kg was confirmed by MS, the other samples were not tested by MS. No relation between concn. of nitrates, nitrites and nitrosamines was observed. These results are discussed in relation to literature data. AJDW

87

Carcinogenicity study on rats fed on canned heated nitrite-treated meat: preliminary communication. (In 'Proceedings of the Second International Symposium on Nitrite in Meat Products' [see FSTA (1977) 9 12S2047].) [Lecture]
Olsen, P.; Meyer, O.
pp. 275-278 (1977) [En] [Inst. of Toxicology, Nat. Food Inst., DK 2860 Soborg, Denmark]

A total of 626 rats was used in a 132 wk, 2-generation feeding trial conducted to evaluate the carcinogenicity of canned chopped pork containing 0-4000 mg nitrite/kg. A control group of rats received a diet based on casein. Data for the incidence of benign and malignant tumours show no significant effect of the nitrite content of the diet on the incidence of either benign or malignant tumours. Dimethylnitrosamine was present in the canned meat containing 4000 mg nitrite/kg, but not in samples containing lower levels of nitrite. AJDW

88

Increased N-nitrosamine formation in model fat systems containing sodium ascorbate. (In 'Proceedings of the Second International Symposium on Nitrite in Meat Products' [see FSTA (1977) 9 12S2047].) [Lecture]
Mottram, D. S.
pp. 135-136 (1977) [En] [ARC Meat Res. Inst., Langford, Bristol B518 1Dy, UK]

Dipropylamine or pyrrolidine (0.125 mmol) dissolved in 100 ml benzene or corn oil was shaken for 2 h at approx. 20°C with 15 ml buffered aqueous solution containing 0.5 mmol sodium ascorbate or solid ascorbyl palmitate, plus 0.25 mmol $NaNO_2$. A control mixture contained no reducing agent. Nitrosamines were extracted by steam distillation, determined by means of an

electrolytic conductivity detector and confirmed by GLC-MS. A table of results is given. The results show that, in this 2-phase system, sodium ascorbate considerably increased nitrosamine formation; ascorbyl palmitate reduced it. This difference is attributable to the sodium ascorbate dissolving in the aqueous phase and reducing NO_2^- to NO, some of which is oxidized to NO_2 and N_2O_3; all 3 nitrogen oxides are powerful nitrosating agents, and rapid nitrosation of the amines occurs in the organic phase. Ascorbyl palmitate is insoluble in water; no reduction of nitrite therefore occurs. These results are discussed in relation to nitrosamine formation in cured meats, e.g. bacon; it is suggested that legislation for compulsory addition of reducing agents to bacon cured is unwise until the mechanism of nitrosamine formation in bacon is fully understood. AJDW

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Specific detection systems for the chromatographic analysis of nitrosamines. (In 'Proceedings of the Second International Symposium on Nitrite in Meat Products' [see FSTA (1977) 9 12S2047].) [Lecture]

Green, C.; Hansen, T. J.; Iwaoka, W. T.; Tannenbaum, S. R.

pp. 145-153 (1977) [En] [Dep. of Nutr. & Food Sci., Massachusetts Inst. of Tech., Cambridge, Massachusetts 02139, USA]

Studies on use of normal-phase and reverse-phase high-pressure liquid chromatography (HPLC) for analysis of a nitrosated spermidine and an extract of raw bacon are described. 3 types of detector were used: (i) a UV detector (254 nm); (ii) a thermal energy analyser specific for N-nitroso compounds; and (iii) a specific photohydrolysis system (coupling the cleaved nitrite to a Griess reagent), and detection of the resulting dye on the basis of its absorbance at 546 nm. Various chromatograms are illustrated. It is concluded that HPLC is highly suitable for separating N-nitroso fractions; the non-destructive nature of HPLC permits collection of samples for further characterization. Selectivities of (ii) and (iii) are similar, and superior to that of (i). Sensitivity of (iii) is <50 ng/injection. HPLC and GLC gave approx. equal values for concn. of nitrosoproline in an extract of raw bacon (70 and 80 µg/kg respectively). Some samples of bacon free from nitrosoproline in the raw state contained nitrosopyrrolidine after frying; this result casts doubt on the significance of pre-formed nitrosoproline as a precursor of nitrosopyrrolidine AJDW

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Extraction, clean-up and estimation as methyl ether of 3-hydroxyl-1-nitrosopyrrolidine, a non-volatile nitrosamine in cooked bacon at mass fractions of µg/kg. (In 'Proceedings of the Second International Symposium on Nitrite in Meat Products' [see FSTA (1977) 9 12S2047].) [Lecture]

Sen, N. P.; Coffin, D. E.; Seaman, S.; Donaldson, B.; Miles, W. F.

pp. 179-185 (1977) [En] [Food Res. Lab., Health Protection Branch, Ottawa, Ontario K1A OL2, Canada]

A method was developed for the estimation of 3-hydroxy-1-nitrosopyrrolidine (HNPyr) in cooked bacon. The sample is extracted with acetonitrile and fats are removed by liquid-liquid extraction with n-heptane. Clean-up is by chromatography on an acidic alumina column. HNPyr is converted to its methyl ether, which is estimated by combined GLC-MS. The limit of detection was about 2 µg/kg with recoveries of 51-102% when spiked with 10-100 µg/kg. Of 13 cooked bacon samples, only 2 contained measurable (8 and 12 µg/kg) mass concn. of HNPyr. AS

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N-nitrosopyrrolidine in bacon obtained from 10 commercial bacon production plants. (In 'Proceedings of the Second International Symposium on Nitrite in Meat Products' [see FSTA (1977) 9 12S2047].) [Lecture]

Birdsall, J. J.

pp. 211-213 (1977) [En] [American Meat Inst., Washington, DC 20007, USA]

Practical trials at 10 commercial bacon factories in the USA are described. Batches of bacon were prepared containing: no $NaNO_2$, erythorbate or ascorbate; or 40, 80 or 120 mg $NaNO_2$/kg together with 550 mg erythorbate or ascorbate/kg. After refrigerated storage for 3 wk, raw samples were analysed for residual nitrite, nitrate and erythorbate; cooked samples were analysed for nitrosopyrrolidine. Of 154 samples studied, only 25 contained presumptive nitrosopyrrolidine by GLC analysis; only 10 were confirmed by MS (1 made with 40 mg nitrite/kg, 4 made with 80 mg/kg and 5 made with 120 mg/kg). The highest nitrosopyrrolidine concn. recorded was 9 mg/kg. Reducing the added $NaNO_2$ concn. from 120 to 80 mg/kg did not markedly appear to reduce the occurrence of low levels (<10 µg/kg) of nitrosopyrrolidine. AJDW

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